

TEST PROCEDURE

TP 204C

Title Gas Analyzer Calibration Curve Generation	Page Number 1 of 22
Originator Nancy Tschirhart	Supersedes TP 204B
Responsible Organization Calibration & Maintenance Group	Computer Program LCS GASCAL
Type of Test Report Computer	Data Form Number NA
Report Distribution Calibration & Maintenance	Implementation Date 9-6-96

Implementation Approval

Original Test Procedure Authorized on 11-14-79

Revision Description

- (1) 9-6-96 The purpose of this change is to revise the procedure as described in EPCN #206. All steps affected by this change are identified with (1) in the margin.

Note: Specific brand names in EPA/EOD procedures are for reference only and are not an endorsement of those products.

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1. Purpose

The purpose of this procedure is to generate analyzer calibration curves used for analysis of exhaust emissions. These curves are then used in the monthly analyzer calibration verifications (TP 303) to assess analyzer curve stability.

A new curve must be generated whenever an existing curve is found to be out of tolerance, when a new analyzer is placed into service, when two or more secondary standard gases have been replaced, or when the top secondary standard gas is named or replaced.

Whenever a new curve is generated the “Calibration Name Activation” program (CNAP) must be run. Refer to Test Procedure 205, “Span Point Change Notice.”

2. Test Article Description

All gas analyzers used for measuring exhaust and evaporative emissions. The following lists the names of the gases and their abbreviations:

<u>Gas Name</u>		<u>Abbreviation</u>		<u>GasCal Abbreviation</u>
Hydrocarbon	=	HC	=	HCAN
Oxides of Nitrogen	=	NO _x	=	NOXA
Carbon Monoxide	=	CO	=	COAN
Carbon Dioxide	=	CO ₂	=	CO2A
Methane	=	CH ₄	=	METH

3. References

- 3.1 “Code of Federal Regulations,” Title 40, Subpart B, 86.121 through 86.124
- 3.2 Laboratory Computer System (LCS) GASCAL user’s manual
- 3.3 “Environmental Protection Agency (EPA) Laboratory Safety Manual”
- 3.4 EPA memo, Subject: “Minor Change to Procedure Description in EPCN 046,” Dave Perkins; October 3, 1983

4. Required Equipment

- 4.1 Calibrated digital voltmeter (DVM) with 0.01-volt resolution or better

- 4.2 Secondary standard calibration gases for the appropriate gas and range being analyzed. See Step 7.13 for the method of selecting the correct gas concentrations.

All secondary gases must have undergone Test Procedure 403, "Gas Correlation," before they may be used as calibration gases.

- 4.3 Portable calibration line of Teflon[®] covered with braided stainless steel for introducing the calibration gases into the analysis system

- 4.4 Active analyzer "Calibration Curve Analysis Book"

5. Precautions

- 5.1 The technician performing the calibration must be familiar with the laboratory safety manual, especially Chapters 2 through 6, which deal with the safe handling of compressed gases.

- 5.2 Moving gas carts may cause injury to feet. Safety shoes should be worn when carts are being moved.

- 5.3 Cylinder carts must not obstruct doorways to the analysis sites. Doorways must remain closed to ensure effectiveness of the fire extinguishing system.

- 5.4 The technician must ensure that there is no leakage of toxic gases and that the analyzer is properly vented to the exhaust ventilation system.

- 5.5 Any time a new curve is generated or updated, a span point change notice must be filed and the new span point posted at that analysis site as soon as the point is known.

No official testing may be done until the new span point is posted. See Attachment A for equipment "tag-out" details.

- (1) 5.6 When processing new curves, the technician will put a line through the curve and write their initials, "Curve Invalid" and the date on the page of the old "Analyzer Calibration Curve Analysis" report for those ranges being changed. The new "Analyzer Calibration Curve Analysis" reports must not be entered into the "Calibration Curve Analysis Book" until the technician verifies that the curves have been updated on Michigan Terminal System (MTS).

6. Visual Inspection

Inspect the portable calibration line for cracks, bends, worn spots, etc.

7. Test Article Preparation

7.1 Verify that the analyzer is operating in accordance with the specifications given in the instruction manual and/or in-house analyzer specification sheet.

7.2 Verify that the analyzer is set in the proper operating configuration:

NO_x analyzer: POWER - "ON"
 NO-NO_x - "NO_x"

Methane analyzer: "CONTINUOUS CYCLE"
 timer switch - "AUTO"
 valve switch - "AUTO"

The methane analyzer toggle switch inside the analyzer must be in the "Auto" mode to run the curve and it must be returned to "Off" when finished.

All other analyzers do not have adjustable configurations.

Ensure that analyzer operating parameters are set correctly. Values for pressure, temperature, and flow rates are posted at the individual sites and on the curve printout.

Refer to the instruction manual and/or the in-house analyzer specification sheet for more detailed instructions, or consult with the team leader.

7.3 Check that the strip chart recorder has sufficient paper and is inking properly.

7.4 Check the calibration label on the calibrated DVM to ensure that the due date has not been exceeded.

If it has, the DVM must be calibrated before the curve generation can proceed.

7.5 If the power is off, allow the strip chart recorder to warm up.

Note: When the strip chart recorder power has been off for any period of time up to 1 hour, an equal period of time with the recorder power on is required for instrument warmup; e.g., a 1-minute power off requires a 1-minute warmup, etc., up to a 1-hour maximum warmup.

If the recorder power has been off for more than 1 hour or if you are unable to determine how long the recorder power has been off, you must allow the recorder to warm up for a minimum of 1 hour. If you turn on a recorder, you must write the "time on" on the chart.

- 7.6 Check the electrical zero of the strip chart by switching the mechanical “ZERO/ON” switch to “ZERO” and seeing that the recorder pen goes to zero on the chart paper. If it does not, adjust it by turning the recorder zero potentiometer for that channel until a zero reading is obtained. Place the “ZERO/ON” switch back in the “ON” position when this has been done.

- 7.7 Attach the calibrated DVM to the analyzer output jack.

- 7.8 Using the on-line working gases, zero and span the instrument on the appropriate multiplier range. Verify that the strip chart recorder reading corresponds to the calibrated DVM reading $\pm 0.2\%$ of full scale.

Verify that the analyzer output noise level is less than $\pm 0.5\%$ of full scale. Noise is defined as short-term cyclical variation of a signal from some average value.

Verify that the analyzer output drift does not exceed $\pm 0.2\%$ of full scale per 2 minutes. Drift is defined as long-term directional change of value.

If any tolerance is exceeded, write on chart paper, “Do not use” and your initials and date. Inform the electronic shop of the problem so that they can make the necessary adjustments.

- 7.9 Record the following data on the strip chart:

Date

Test site number

Operator’s name and ID number

Gas analyzed

Standard laboratory range

Analyzer property ID number

Sample flow rate

Air pressure (FID)

Fuel pressure (FID)

Sample pressure (FID)

Barometer reading for CO₂ calibrations

See Attachment B for an example of the strip chart

- 7.10 At analysis sites where a calibration port is provided, turn off the span gas flow to the untested analyzers by selecting a blank “SPAN” button.

Switch the analyzer being used to the “OFF” mode and select blank “SPAN” buttons for the on-site analyzers not being tested. This prevents waste of calibration gases.

At analysis sites where a calibration port is not provided, switch the analyzer to the span mode and plug the secondary gas into the span port. When this has been done, plug the span gas line back into the port.

- 7.11 Select the proper secondary standards to be used as data points in the curve.

The curve should include those cylinders whose concentrations will produce the following approximate deflection readings at the range being calibrated:

Nonlinear Analyzers (NDIR)
minimum of eight data points

Approximate Chart
% Full Scale

95
80
70
60
50
40
25
15

Linear Analyzers (FID, HFID, Chemil, GC)
minimum of six data points

Approximate Chart
% Full Scale

90
75
60
45
30
15

If not enough gases are available to meet these requirements, consult the team leader for further action. More gases may be used in the curve to provide quality control data on gas concentration uniformity.

All secondary standards must have blue EPA labels showing the Gas Lab-named concentration.

8. Test Procedure**100 Generate Curve Data**

101 Using the portable calibration line, connect and open the highest concentration secondary gas cylinder to be used in the curve to the appropriate instrument gas input port.

102 Adjust the instrument gas flow rate and pressure as closely as possible to the rates posted at the analysis site.

Continue to monitor them throughout the procedure.

103 Zero the instrument using the on-line zero gas. Adjust the zero potentiometer on this instrument until a stable and accurate zero reading is obtained. Label this area with a 0 (zero).

Note: A stable reading is defined as 1 minute of measurement in which the drift variation is not more than $\pm 0.2\%$ of full scale from the set point and the noise variation is not more than $\pm 0.5\%$ of full scale from that same set point.

The numerical value of the reading is the operator's estimate of the average reading occurring during the measurement period. The calibrated DVM is used for all measurements.

104 Span the instrument using the highest concentration secondary gas. Adjust the span potentiometer until the DVM reading equals the percentage of the full scale of the range being analyzed that corresponds to the highest concentration secondary gas. Label this area with the letters "TB," which stands for top bottle.

Example percentage full scale calculation:

The range is 0-250 ppm and the highest concentration secondary gas is 230 ppm; adjust the DVM reading to 92% of full scale (230 ppm = 92% of 250 ppm).

The span reading from the previous curve may be used again if the highest concentration secondary gas cylinder has not been renamed or replaced.

105 Zero check the instrument and allow the reading to stabilize. Label this area with a 0 (zero).

If the reading does not return to zero within $\pm 0.3\%$ of full scale, adjust the potentiometer until it does and repeat Steps 103 and 104 until the reading is within tolerance.

- 106 Span the instrument using the highest concentration secondary gas cylinder and allow the reading to stabilize without adjusting the potentiometer. Label this area with the letters "TB."
- If the DVM reading does not match the original span reading obtained in Step 104 within $\pm 0.3\%$ of full scale, repeat Steps 103-105.
- 107 Run the curve. In descending order of concentration, introduce the sequence of secondary calibration gases to be used as data points in the curve. Obtain and record a stable DVM reading for each without adjusting the potentiometer.
- For each reading taken, write the cylinder number, the concentration from the EPA Gas Lab label attached to the cylinder, and the observed DVM reading on the strip chart near the measurement area. Circle the DVM reading.
- 108 Span check the instrument by introducing the highest concentration secondary gas (top bottle) for a reference span point. Label this area with the letters "TB."
- If the span check reading has drifted more than $\pm 0.3\%$ of full scale from the reading obtained in Step 106, repeat Steps 101-107 until all the requirements are met.
- If unable to obtain a span check within the tolerance, the drift problem must be corrected before the curve can be completed.
- The analyzer range will be "tagged out" of service until it can be deactivated on LCS and the corresponding span point will be crossed out on the analyzer bench. Notify the appropriate testing group of any analyzer range that is deactivated.
- 109 If the top bottle check reading is not exactly the value obtained in Step 104 but it is within $\pm 0.3\%$ full scale from that value, adjust the potentiometer to correspond to the highest concentration secondary gas.
- 110 Introduce the working span gas and obtain a stable reading. Record the reading on the strip chart where it occurs, along with the actual concentration and gas cylinder number. This step provides information for TP 205, Span Point Change Notice.
- 111 Zero check the instrument using the on-line zero gas. Do not adjust the potentiometer.
- If the zero point has drifted more than $\pm 0.3\%$ of full scale, repeat Steps 101-110.
- If the zero point is still out of tolerance, the drift problem must be corrected before the curve can be completed.

112 After completing all the above steps, place all span gas buttons in the “OFF” position.

113 Turn off all secondary gas bottles and disconnect them and any other related equipment. If needed, connect the span cylinders. Verify that the flow rate is set to the required rate posted for each instrument.

- (1) 114 If processing a new curve write your initials, “Curve Invalid” and the date on the page of the old “Analyzer Calibration Curve Analysis” report for the range(s) changed.

When you receive the MTS “SAQR” report(s), verify that the “Equipment ID#” and coefficients correspond to those on the “Analyzer Calibration Curve Analysis” report. This verifies that the curve(s) was(were) updated on MTS by Computer Operations. If they correspond correctly, sign your name at the bottom of the report. Remove the old copy of the curve(s) from the “Calibration Curve Analysis Book” and put the new curve in its place.

(1) **200 Interpolate Curve Data**

If only one secondary standard gas, other than the top bottle, has been replaced, the curve point must be interpolated.

201 Use the “Curve Table” and locate the value above and below the named concentration of the secondary bottle.

202 Subtract the lower value from the upper value.

203 Subtract the named secondary concentration from the lower value.

204 Divide the result from Step 203 by the result from Step 202. This will give you a number of 10 ths.

205 Add the number obtained in Step 204 to the “Deflection Value” of the corresponding lower value. This becomes the secondary gas calculated curve point.

206 Compare the secondary gas calculated curve point to the actual value obtained from the analyzer. The secondary gas calculated curve point must be within $\pm 1.0\%$ of the actual value, otherwise the bottle must be re-analyzed on the Master Analysis Bench or replaced.

If the reading is within $\pm 1.0\%$, record the value in the “Calibration Curve Analysis Book” and put your initials next to it.

9. Data Input

- 9.1 The operator writes the cylinder number, the concentration from the EPA Gas Lab label attached to the cylinder, and the observed DVM reading on the strip chart near the measurement area.

Zero and top bottle span points are indicated as such. The on-line working gas is identified as "WG."

All DVM readings are circled.

- 9.2 The data is entered interactively from an LCS terminal using the on-line "Gas Analyzer Calibration" program (GASCAL). Do not type the quote marks for any of the following typed entries.

- 9.2.1 To retrieve the most current calibration data set from the "Input Save" file:

Locate an LCS terminal and access the program by pressing <BREAK>, then type "\$RUN GASCAL" and press <RETURN>.

Type "USE" and press <RETURN>. The program will respond and request information to identify the calibration data set.

Type in the analyzer ID# (EPA property ID#) and press <RETURN>.

Type in the gas name and range and press <RETURN>.

Type in the data set number and press <RETURN>.

Type in the calibration set number and press <RETURN>.

See Attachment C for an example of this data input.

- 9.2.2 To get into the edit mode to change data:

Type in "OVERLAY CARDS" and press <RETURN>.

Each card will print out one line at a time so you can make the necessary corrections. Make all changes to the card by typing in the new data, overlaying the current data.

See Attachment D for an example.

Note: A typing shortcut for "OVERLAY CARDS" is "OV CARDS."

9.2.3 To verify that the changes have been made, type “LIST CARDS” and press <RETURN>. See Attachment E for an example.

If no corrections are required, type “CALC” and press <RETURN>. This will initiate the calibration calculations.

9.2.4 Type “PRINT P2” and press <RETURN>. This will print the “Analyzer Calibration Curve Analysis” for verification that the data meet the acceptance criteria. See Attachment F for an example of this report.

9.2.5 If the data are within the limits, type “LINEPRINT” and press <RETURN>.

Enter “3” for the number of copies required and press <RETURN>. Retrieve the printouts from the computer operations pick-up window.

9.2.6 Type in “SAVE” and press <RETURN>. The terminal will print out the following:

Analyzer ID: 123344
Calibration Name: Gas Name
Data Set Number:
Calibration Number:

OK TO SAVE DATA WITH THESE IDENTIFIERS? (Y/N/CTLC)

If OK, type “Y” and press <RETURN>.

Type “TER” and press <RETURN> to terminate the program.

9.3 Refer to Test Procedure 205, “Span Point Change Notice” to initiate the “Calibration Name Activation” program (CNAP) to obtain new span points.

10. Data Analysis

10.1 The technician examines the “Analyzer Calibration Curve Analysis” report and strip charts for each analyzer range and determines if the curve meets the acceptance criteria.

10.2 If the curve does not meet the acceptance criteria, refer to Attachment G, “Troubleshooting Flowchart for Invalid Analyzer Curves,” for corrective measures.

- 10.3 If inflection points are flagged on the “Analyzer Calibration Curve Analysis” report, they must be investigated by Calibration & Maintenance (C&M) before the curve is accepted. A curve fit of more than 0.5% on the lowest secondary gas cylinder indicates a null offset optimization problem.

On the Low CO, High CO, or CO₂ “Analyzer Calibration Curve Analysis” reports, in the “Curve Comparisons” box, under the “Y %POINT (M C)/C” header, ensure that the value the “MEAN of (ABS%)” is less than 0.5%. If it is not, notify the C&M manager that there is a null offset optimization problem with the analyzer.

- (1) 10.4 When processing new curves, put a line through the curve and write your initials, “Curve Invalid” and the date on the page of the old “Analyzer Calibration Curve Analysis” report for those ranges being changed.
- (1) 10.5 The technician obtains the MTS “SAQR” report(s) and verifies that the equipment ID# and coefficients correspond to those on the “Analyzer Calibration Curve Analysis” report(s). If they do, they will remove the old “Analyzer Calibration Curve Analysis” report(s), sign the bottom, and put the new report(s) into the “Calibration Curve Analysis Book.”
- (1) 10.6 One secondary gas may be replaced without running a new curve by interpolating a response off the existing curve. The required response, cylinder number, and concentration must be written on the “Analyzer Calibration Curve Analysis” report.

11. Data Output

- 11.1 The “Analyzer Calibration Curve Analysis” report is printed for each analyzer range.
- (1) 11.2 Computer Operation prints the “SAQR” report is printed for each analyzer range. The report is given to C&M for confirmation that the curves have been updated on MTS.
- 11.3 Copies of the “Analyzer Calibration Curve Analysis” report are distributed as follows:
- Deliver one copy to computer operations for update purposes.
 - File one copy in the C&M active curve file, replacing the old curve if applicable. File the old curve in the inactive file.
 - File one copy in a file at the analyzer site. Remove and destroy the record of the old curve.
- 11.4 The strip chart is kept in C&M under the analyzer site number and date of completion.

12. Acceptance Criteria

- 12.1 All zero and span checks must be within $\pm 0.3\%$ full scale of the original readings.
- 12.2 For the curve to be valid, all figures in the column under “curve fit deviation” marked “% point” must be within $\pm 1\%$.
- 12.3 The “average deviation” found at the bottom of this section must not be more than $\pm 0.5\%$.
- 12.4 At least eight data points must be used in curves for nonlinear analyzers. At least six data points must be used in curves for linear analyzers.
- 12.5 The percent non linearity must not be more than 10% for all analyzers except NDIRs. If the non linearity of an NDIR exceeds 15%, it is investigated by the team leader before the curve is accepted.
- (1) 12.6 The curve coefficients on the “SAQR” report correspond to the values on the “Analyzer Calibration Curve Analysis” report.

13. Quality Control Provisions

- 13.1 All analytical instruments must be properly warmed up and in a test-ready mode prior to use.
- 13.2 All DVMs used in the procedure must have undergone a routine calibration within the past 90 days.
- 13.3 Percent deviations should be random with respect to \pm signs. With approval of the C&M manager, nonstandard ranges may require higher degree fit to resolve this problem. Consult with the C&M manager in such cases.
- 13.4 The curves are generated mathematically using the following:
 - Hydrocarbon ---third order equation
 - Oxides of Nitrogen ---second order equation
 - Carbon Monoxide ---third order equation
 - Carbon Dioxide ---third order equation
 - Methane ---second order equation

13.5 If an instrument can not be calibrated because of performance problems, it will be tagged as inoperative and deactivated as described in Attachment A.

- (1) 13.6 A technician that did not run the curve will compare the equipment ID # and the coefficients on the “SAQR” report to those on the “Analyzer Calibration Curve Analysis” report to ensure consistent data processing from both LCS and MTS systems.

Instrument Tag-Out Process

If any equipment calibration can not be successfully completed or if the equipment is otherwise determined to be unusable, it must be “tagged out” of service to prevent it being used in the test process. When all acceptance criteria have been met, the tag will be removed and the equipment will be available for use in the test process.

If an analyzer is not calibrated or it is otherwise determined unusable, the analyzer site (i.e., A001, A002, or A003) will be “tagged out” of service until the problem is resolved. When all acceptance criteria have been met for the analyzer, the tag will be removed and the site will be available for use.

If only one range of an analyzer is not calibrated or that range is otherwise determined unusable, unless the problem can be rectified within 3 hours, that range will be “tagged out” of service until it is deactivated on LCS to prevent it from being used in the test process. When all acceptance criteria have been met for the analyzer range, the range will be activated on LCS and it will be available for use in the test process.

Equipment that is tagged out of service will be documented in the FSB Log and recorded on the “Equipment Status” board between SHEDs 1 and 2.

The tag will be attached to the equipment as follows:

- Strip chart - taped to the front panel

- Analyzer - hung on the flow control knob for that instrument

- Dynamometer - hung on “Inertia Weight” selector knob

- Constant Volume Sampler - hung on “Vehicle Test” selector knob

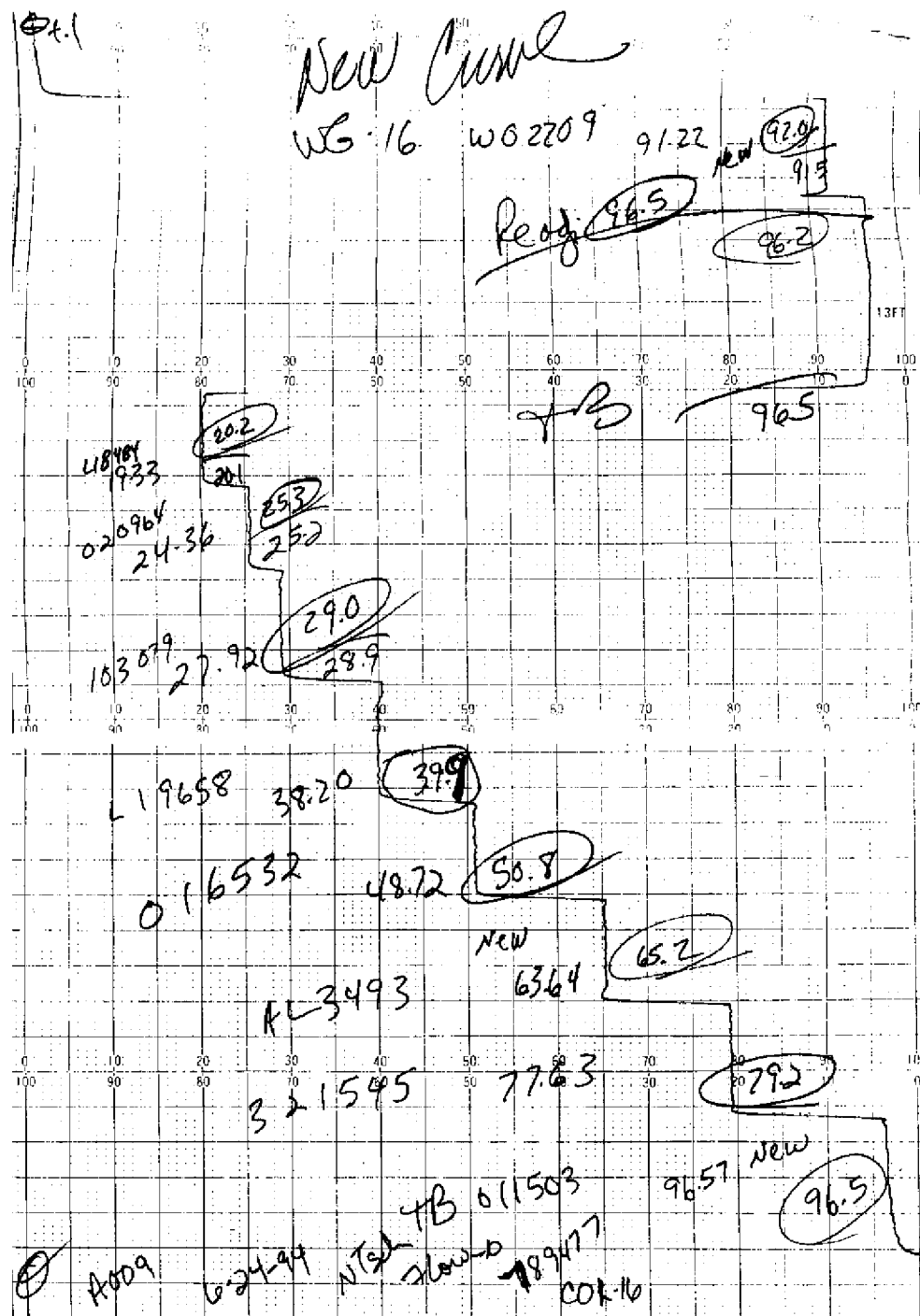
- Miscellaneous equipment - available location

Each tag will contain the following information:

- Equipment Name

- Date and Time

- Technician Name



T.S.P. OPCOM X PROD

??\$RUN GASCAL

GASCAL09 BEGINS EXECUTION.

VERSION 1.1:02/28/85

06/24/94 07:32:01

C USE

ENTER DATA SET IDENTIFIERS (OR CTRL-C TO CANCEL OR CORRECT):

1. ENTER ANALYZER ID : 707477
2. ENTER CALIBRATION NAME : COAN CR16
3. ENTER DATA SET NUMBER :
4. ENTER CALIBRATION NUMBER:

C-0V CARDS

GROUP NAME: CARDS

```

: TA 709477 A007 16 CO N2 PPM 13900 0100 101593 1 1 3 2 X 5
: TA 709477 A007 16 CO N2 PPM 36557 3 062424
: TD DVM HORIDA 3.0 SCFH 0.0 22 0.0 0.0 0.0
: TD DVM HORIDA 10.0 SCFH 0.0 22 0.0 0.0 0.0
: TC 0-100 PPM CO NEW HORIDA ANALYZER 3/6/89
: TC 0 100 PPM CO NEW HORIDA ANALYZER 3/6/89
:
:
:
:
:

```

GROUP NAME: DATA CARDS

```

DATA CARD 01 : 01 207253 X 94.91 94.9
: 011503 : 6.57 6.5
DATA CARD 01 : 01 011503 X 96.57 96.5
DATA CARD 02 : 02 321545 X 77.63 70.9
: : 7.2
: 02 321545 X 77.63 77.2
: 03 016701 X 64.33 65.9
: AL3493 : 3.64 2
DATA CARD 03 : 03 AL3493 X 63.64 65.2
DATA CARD 04 : 04 016532 X 48.72 50.5
: : 0
DATA CARD 04 : 04 016532 X 48.72 50.8
DATA CARD 05 : 05 L17650 X 30.20 39.7
: : 9
DATA CARD 05 : 05 L17650 X 38.20 39.9
DATA CARD 06 : 06 103079 X 27.92 28.9
: : 9.0
DATA CARD 06 : 06 103079 X 27.92 29.0
DATA CARD 07 : 07 020964 X 24.36 25.2
: : 3
DATA CARD 07 : 07 020964 X 24.36 25.3
DATA CARD 08 : 08 L10404 X 19.33 20.1
: : 2
DATA CARD 08 : 08 L10404 X 19.33 20.2
DATA CARD 09 : 09 W02140 X 09.67 90.0
: : 209
DATA CARD 09 : 09 W02209 X 91.22 92.0
DATA CARD 10 : 10 ZERO X 0.0 0.0

```

C LIST CARDS

GROUP NAME: CARDS

TA CARD : TA 782477 A002 16 CO N2 PPM 36557 0600 062494 1 1 3 2 X 5

TB CARD : TB DVM HORIDA 10.0 SCFH 0.0 22 0.0 0.0 0.0

TC CARD : TC 0-100 PPM CO NEW HORIDA ANALYZER 3/6/89

GROUP NAME: DATA CARDS

DATA CARD 01	: 01 011503	X	96.57	96.5
DATA CARD 02	: 02 321545	X	77.63	79.2
DATA CARD 03	: 03 4L3493	X	63.64	65.2
DATA CARD 04	: 04 016532	X	40.72	50.8
DATA CARD 05	: 05 L19650	X	30.20	39.9
DATA CARD 06	: 06 103079	X	27.92	29.0
DATA CARD 07	: 07 020964	X	24.36	25.3
DATA CARD 08	: 08 L10404	X	19.33	20.2
DATA CARD 09	: 09 W02209	X	21.22	22.0
DATA CARD 10	: 10 ZERO	X	0.0	0.0
DATA CARD 11	:			
TZ CARD	: TZ :			

Troubleshooting Flowchart for Invalid Analyzer Curves